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<b>(54) Title:</b> COATED ENCAPSULATED ACTIVE INGREDIENTS, CHEWING GUMS CONTAINING SAME, AND METHODS OF PREPARING SAME  <b>(57) Abstract</b>  The present invention provides encapsulated sensitive active ingredients, such as aspartame, and chewing gum including same. Pursuant to the present invention, a sensitive active ingredient, such as aspartame, that is coated with polyvinyl acetate, is coated with a second coating of zein. The zein coating protects the polyvinyl acetate encapsulation thereby protecting the sensitive active ingredient from degradation.		

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- 1 -

S P E C I F I C A T I O NTITLE

"COATED ENCAPSULATED ACTIVE INGREDIENTS,  
CHEWING GUMS CONTAINING SAME, AND METHODS  
OF PREPARING SAME"

BACKGROUND OF THE INVENTION

5 The present invention relates generally to chewing  
gums and the manufacture of same. More specifically, the  
present invention relates to chewing gums having  
10 ingredients that must be encapsulated in order to prevent  
degradation.

It is, of course, known to prepare chewing gum  
including a variety of different ingredients. Some of  
these ingredients are used to add flavor or sweetness to  
15 the chewing gum. One of the difficulties encountered in  
adding certain ingredients to chewing gum is that they  
are vulnerable to degradation.

It is known, to add aspartame, a sugarless  
sweetener, to chewing gum. However, aspartame is  
20 vulnerable to degradation; upon exposure to moisture  
aspartame will degrade. Therefore, it has been known to  
encapsulate aspartame in polyvinyl acetate to prevent  
degradation. In a similar vein, other sensitive  
ingredients have been encapsulated with polyvinyl acetate  
25 to prevent degradation, for example, alitame. U.S.  
Patent No. 5,108,763 discloses the use of polyvinyl  
acetate to encapsulate sweeteners.

Unfortunately, even when encapsulated in polyvinyl  
acetate, aspartame is still vulnerable to degradation.  
30 In this regard, flavor components present in the chewing  
gum can plasticize the polyvinyl acetate coating. This  
can destroy or reduce the protective effects of the

- 2 -

polyvinyl acetate, rendering the aspartame, or other active sensitive ingredient, vulnerable to degradation.

For example, it is known that polyvinyl encapsulations are not stable in certain chewing gums such as fruit flavored gums. This is because the fruit flavor in the chewing gum plasticizes the polyvinyl acetate coating of the active ingredient and compromises the integrity of the encapsulated particle.

A number of patents disclose methods of coating ingredients, such as aspartame. These patents include U.S. Patent Nos: 4,863,745; 4,919,941; 4,931,295; 5,108,762; 5,124,160; 5,139,798; 5,154,939; 5,164,210; 5,165,944; 5,169,657; 5,169,658; 5,112,625; 5,192,563; 5,198,251; 5,364,627; and European Patent No. 484,124. Other patents disclosing the use of coating materials include U.S. Patent Nos. 5,077,053; 4,384,004; 2,504,436; 3,576,663; 4,137,300; 4,194,013; 4,224,219; and 4,474,749. See also: French Patent No. 2,117,506; and Japanese Patent Nos. 82-141273 and 88-028575.

There is still, however, a need for an improved method of encapsulating sensitive active ingredients, such as aspartame, especially in fruit flavored chewing gums.

#### SUMMARY OF THE INVENTION

The present invention provides encapsulated sensitive active ingredients, such as aspartame, and chewing gum including same. Pursuant to the present invention, a sensitive active ingredient, such as aspartame, that is coated with polyvinyl acetate, is coated with a second coating of zein. The zein coating protects the polyvinyl acetate encapsulation thereby

- 3 -

protecting the sensitive active ingredient from degradation.

To this end, a chewing gum is provided including a water insoluble portion, one or more flavors, and a water  
5 soluble portion. The chewing gum also includes a sensitive active ingredient coated with a first coating of polyvinyl acetate and a second coating of zein that coats the first coating.

In an embodiment, the active ingredient is chosen  
10 from the group consisting of aspartame, alitame, sucralose, and glycyrrhizin.

In an embodiment, the flavor is chosen from the group consisting of amyl acetate, lemon oil, orange oil, ethyl butyrate, ethyl propionate, ethyl acetate, ethyl  
15 alcohol, ethyl caproate, and aldehyde flavors.

In an embodiment, the second coating is applied at a weight ratio of approximately 1:10 to about 1:1 as compared to a total cumulative weight of the first coating and sensitive active ingredient.

20 In an embodiment, the second coating is applied by wet granulation.

In another embodiment, an encapsulated sensitive active ingredient is provided comprising: a sensitive active ingredient; a first coating of polyvinyl acetate  
25 that coats at least a substantial portion of an outer surface of the sensitive active ingredient; and a second coating of zein that coats at least a substantial portion of an outer surface of the first coating.

In still another embodiment, a method for preparing  
30 chewing gum is provided comprising the steps of: coating a sensitive active ingredient with polyvinyl acetate to create an encapsulated product; and coating the encapsulated product with a solution chosen from the

- 4 -

group consisting of zein, gelatin, and water soluble proteins.

5 In an embodiment, the sensitive active ingredient is coated with polyvinyl acetate through wet granulation, wax granulation, spray drying, spray chilling, fluid bed coating, coacervation, extrusion, or fiber spinning.

An advantage of the present invention is to provide an improved chewing gum composition.

10 Still further, an advantage of the present invention is to provide an improved method of encapsulating sensitive active ingredients.

15 Moreover, an advantage of the present invention is to provide sensitive active ingredients that are encapsulated so as to prevent degradation of the ingredients.

Furthermore, an advantage of the present invention is to provide an improved method for protecting sensitive active ingredients in chewing gum compositions especially those including fruit flavor.

20 Another advantage of the present invention is to provide an improved method for manufacturing chewing gum.

Still further, an advantage of the present invention is to provide an improved method for protecting sweeteners from degradation in chewing gum.

25 Further, an advantage of the present invention is to provide an improved method for encapsulating moisture sensitive ingredients.

30 Additional features and advantages of the present invention are described in, and will be apparent from, the detailed description of the presently preferred embodiments and from the Figure.

- 5 -

### BRIEF DESCRIPTION OF THE FIGURE

The Figure illustrates graphically the results of the experiments set forth as Examples 1-3 below.

5

### DETAILED DESCRIPTION

#### OF THE PRESENTLY PREFERRED EMBODIMENTS

The present invention provides an improved method for encapsulating sensitive active ingredients, such as aspartame and alitame. Pursuant to the present invention, the sensitive active ingredients are encapsulated by a first coating of polyvinyl acetate. The polyvinyl acetate coating is then encapsulated by, preferably, a coating of zein. By so encapsulating the polyvinyl acetate coating, the coating of polyvinyl acetate is protected from degradation, and specifically from being plasticized by flavors such as fruit. This therefore protects the polyvinyl acetate encapsulation of the sensitive active ingredient. It should also be noted that it may be desirable to coat an ingredient to provide longer flavor or sweetness and thereby control the release of the ingredient.

There are a number of methods of encapsulating aspartame, or like sensitive active ingredients, with polyvinyl acetate. Any such method can be used pursuant to the present invention including wet granulation, wax granulation, spray drying, spray chilling, fluid bed coating, coacervation, extrusion or fiber spinning. Preferably, the ratio of sensitive active ingredient, e.g., aspartame, to polyvinyl acetate when so coated is preferably from approximately 1:9 to about 1:4.

As used herein, "sensitive active ingredient" refers to any ingredient that can be used in a confectionery or food product that upon exposure to the ambient

- 6 -

environment, or other ingredients in the product, will degrade and not fully provide its desired properties or characteristics. Aspartame and alitame are examples of sensitive active ingredients. The present invention can  
5 be used to protect such sensitive active ingredients.

To this end, in an embodiment, the present invention provides a coating of zein that is used to coat a layer of polyvinyl acetate that surrounds the sensitive active ingredient, such as aspartame. Although preferably zein  
10 is used to encapsulate the polyvinyl acetate coating, if desired other compositions can be used instead of zein such as gelatin and water soluble proteins such as casein, soy and glutan.

Pursuant to the present invention, in an embodiment, once the sensitive active ingredient, e.g., aspartame, is encapsulated by the polyvinyl acetate, it is then coated with an aqueous zein solution. The zein solution is made by dispersing approximately 5 to about 20% by weight powdered zein in approximately 80 to about 95% by  
15 weight water. Preferably, the resultant product has a pH of approximately 8 to about 10.5.

During the process, in order to dissolve the zein, the pH of the dispersion is increased to approximately 11.6 to about 11.9. The zein solution is then in a  
20 condition that it can be applied to the polyvinyl acetate encapsulated aspartame by a number of methods including wet granulation.

The zein solution is applied so that, preferably, the ratio of zein to the polyvinyl acetate encapsulated sensitive active ingredient is from about 1:10 to about  
25 1:1.

In creating a chewing gum product, the zein overcoated polyvinyl acetate coated sensitive active



- 7 -

ingredients can be added so that the resultant chewing gum product comprises approximately 0.01 to about 3% by weight of the overcoated ingredients. Most preferably, the overcoated ingredients are added at a ratio of approximately 0.1 to about 1.5% and in a preferred embodiment, approximately 0.3 to about 0.8%.

There are a variety of different gums that the zein overcoated polyvinyl acetate coated sensitive active ingredients can be used in. In a preferred embodiment, the overcoated ingredients will be used in fruit flavored chewing gum.

There are a variety of fruit flavors that are used in chewing gums, such as, amyl acetate, lemon oil, orange oil, ethyl butyrate, ethyl propionate, ethyl acetate, ethyl alcohol and ethyl caproate. The zein overcoated ingredient could also be used in chewing gums containing aldehyde flavors such as cinnamic aldehyde to protect the aspartame from being degraded by the aldehyde. Additional (non-fruit) flavors may optionally be included such as peppermint oil, spearmint oil, other mint oils, clove oil, oil of wintergreen, and anise. The flavor is generally present in the gum at approximately 0.3 to about 10% by weight of the gum.

The chewing gum that utilizes the present invention may be any of a variety of different chewing gums, including low or high moisture, sugar or sugarless, wax-containing or wax-free, low calorie and/or a chewing gum that includes dental health agents.

Chewing gum generally consists of a water insoluble gum base, a water soluble portion, and flavors. The water soluble portion dissipates with a portion of the flavor over a period of time during chewing. The gum

- 8 -

base portion is retained in the mouth throughout the chew.

5 The insoluble gum base generally comprises elastomers, resins, fats and oils, softeners, and inorganic fillers. The gum base may or may not include wax. The insoluble gum base can constitute approximately 5 to about 95 percent, by weight, of the chewing gum, more commonly, the gum base comprises 10 to about 50 percent of the gum, and in some preferred embodiments, 10 20 to about 35 percent, by weight, of the chewing gum.

In an embodiment, the chewing gum base of the present invention contains about 20 to about 60 weight percent synthetic elastomer, 0 to about 30 weight percent natural elastomer, about 5 to about 55 weight percent elastomer plasticizer, about 4 to about 35 weight percent 15 filler, about 5 to about 35 weight percent softener, and optional minor amounts (about one percent or less) of miscellaneous ingredients such as colorants, antioxidants, etc.

20 Synthetic elastomers may include, but are not limited to, polyisobutylene with a GPC weight average molecular weight of about 10,000 to about 95,000, isobutylene-isoprene copolymer (butyl elastomer), styrene-butadiene copolymers having styrene-butadiene ratios of about 1:3 to about 3:1, polyvinyl acetate 25 having a GPC weight average molecular weight of about 2,000 to about 90,000, polyisoprene, polyethylene, vinyl acetate-vinyl laurate copolymer having vinyl laurate content of about 5 to about 50 percent by weight of the copolymer, and combinations thereof. 30

Preferred ranges are, for polyisobutylene, 50,000 to 80,000 GPC weight average molecular weight, for styrene-butadiene, 1:1 to 1:3 bound styrene-butadiene,

- 9 -

for polyvinyl acetate, 10,000 to 65,000 GPC weight average molecular weight with the higher molecular weight polyvinyl acetates typically used in bubble gum base, and for vinyl acetate-vinyl laurate, vinyl laurate content of 10-45 percent.

Natural elastomers may include natural rubber such as smoked or liquid latex and guayule as well as natural gums such as jelutong, lechi caspi, perillo, sorva, massaranduba balata, massaranduba chocolate, nispero, rosindinha, chicle, gutta hang kang, and combinations thereof. The preferred synthetic elastomer and natural elastomer concentrations vary depending on whether the chewing gum in which the base is used is adhesive or conventional, bubble gum or regular gum, as discussed below. Preferred natural elastomers include jelutong, chicle, sorva and massaranduba balata.

Elastomer plasticizers may include, but are not limited to, natural rosin esters such as glycerol esters of partially hydrogenated rosin, glycerol esters of polymerized rosin, glycerol esters of partially dimerized rosin, glycerol esters of rosin, pentaerythritol esters of partially hydrogenated rosin, methyl and partially hydrogenated methyl esters of rosin, pentaerythritol esters of rosin; synthetics such as terpene resins derived from alpha-pinene, beta-pinene, and/or d-limonene; and any suitable combinations of the foregoing. the preferred elastomer plasticizers will also vary depending on the specific application, and on the type of elastomer which is used.

Fillers/texturizers may include magnesium and calcium carbonate, ground limestone, silicate types such as magnesium and aluminum silicate, clay, alumina, talc, titanium oxide, mono-, di- and tri-calcium phosphate,

- 10 -

cellulose polymers, such as wood, and combinations thereof.

5 Softeners/emulsifiers may include tallow, hydrogenated tallow, hydrogenated and partially hydrogenated vegetable oils, cocoa butter, glycerol monostearate, glycerol triacetate, lecithin, mono-, di- and triglycerides, acetylated monoglycerides, fatty acids (e.g. stearic, palmitic, oleic and linoleic acids), and combinations thereof.

10 Colorants and whiteners may include FD&C-type dyes and lakes, fruit and vegetable extracts, titanium dioxide, and combinations thereof.

15 The base may or may not include wax. An example of a wax-free gum base is disclosed in U.S. Patent No. 5,286,500, the disclosure of which is incorporated herein by reference.

20 In addition to a water insoluble gum base portion, a typical chewing gum composition includes a water soluble bulk portion and one or more flavoring agents. The water soluble portion can include bulk sweeteners, high intensity sweeteners, flavoring agents, softeners, emulsifiers, colors, acidulants, fillers, antioxidants, and other components that provide desired attributes.

25 Softeners are added to the chewing gum in order to optimize the chewability and mouth feel of the gum. The softeners, which are also known as plasticizers and plasticizing agents, generally constitute between approximately 0.5 to about 15% by weight of the chewing gum. The softeners may include glycerin, lecithin, and combinations thereof. Aqueous sweetener solutions such as those containing sorbitol, hydrogenated starch hydrolysates, corn syrup and combinations thereof, may

30

- 11 -

also be used as softeners and binding agents in chewing gum.

Bulk sweeteners include both sugar and sugarless components. Bulk sweeteners typically constitute 5 to  
5 about 95% by weight of the chewing gum, more typically, 20 to 80% by weight, and more commonly, 30 to 60% by weight of the gum.

Sugar sweeteners generally include saccharide-containing components commonly known in the chewing gum  
10 art, including, but not limited to, sucrose, dextrose, maltose, dextrin, dried invert sugar, fructose, levulose, galactose, corn syrup solids, and the like, alone or in combination.

Sugarless sweeteners include, but are not limited  
15 to, sugar alcohols such as sorbitol, mannitol, xylitol, hydrogenated starch hydrolysates, maltitol, and the like, alone or in combination.

High intensity artificial sweeteners can also be used, alone or in combination with the above. Preferred  
20 sweeteners include, but are not limited to sucralose, aspartame, salts of acesulfame, alitame, saccharin and its salts, cyclamic acid and its salts, glycyrrhizin, dihydrochalcones, thaumatin, monellin, and the like, alone or in combination.

Usage level of the artificial sweetener will vary greatly and will depend on such factors as potency of the  
25 sweetener, rate of release, desired sweetness of the product, level and type of flavor used and cost considerations. Thus, the active level of artificial sweetener may vary from 0.02 to about 8%. When carriers  
30 used for encapsulation are included, the usage level of the encapsulated sweetener will be proportionately higher.

- 12 -

Combinations of sugar and/or sugarless sweeteners may be used in chewing gum. Additionally, the softener may also provide additional sweetness such as with aqueous sugar or alditol solutions.

5 Flavor should generally be present in the chewing gum in an amount within the range of about 0.1-15% by weight of the chewing gum, preferably between about 0.2-5% by weight of the chewing gum, mostly preferably between about 0.5-3% by weight of the chewing gum.

10 Flavoring agents may include essential oils, synthetic flavors or mixtures thereof including, but not limited to, oils derived from plants and fruits such as citrus oils, fruit essences, peppermint oil, spearmint oil, other mint oils, clove oil, oil of wintergreen, anise and

15 the like. Artificial flavoring agents and components may also be used in the flavor ingredient of the invention. Natural and artificial flavoring agents may be combined in any sensorially acceptable fashion.

Optional ingredients such as colors, emulsifiers, pharmaceutical agents and additional flavoring agents may

20 also be included in chewing gum.

Chewing gum is generally manufactured by sequentially adding the various chewing gum ingredients to any commercially available mixer known in the art.

25 After the ingredients have been thoroughly mixed, the gum mass is discharged from the mixer and shaped into the desired form such as by rolling into sheets, scoring and cutting into pieces. Generally, the ingredients are mixed by first melting the gum base and adding it to the running mixer. The gum base may alternatively be melted

30 in the mixer. Color and emulsifiers can be added at this time.

- 13 -

A softener such as glycerin can be added next along with syrup and part of the bulk portion. Further, parts of the bulk portion may then be added to the mixer. Flavoring agents are typically added with the final part  
5 of the bulk portion. The entire mixing process typically takes from five to fifteen minutes, although longer mixing times are sometimes required. Those skilled in the art will recognize that variations of this mixing procedure, or other mixing procedures, may be followed.

10 By way of example, and not limitation, examples of the present invention will now be given.

EXAMPLE 1

Aspartame was obtained in the form of a fine, white powder and placed into fruit flavored chewing gum at  
15 0.10% by weight of the gum. The gum formula is set forth below in Table 1 as Example 1.

EXAMPLE 2

A mixture of aspartame and polyvinyl acetate (PVAc) was melt spun into fibers in an extruder. After the  
20 fibers were formed, they were stretched by applying a stretching force such as a draw. The fibers were then cut and placed into fruit flavored chewing gum at 0.5% by weight of the gum. The gum formula is set forth below in Table 1 as Example 2.

25 EXAMPLE 3

The polyvinyl acetate encapsulated aspartame of Example 2 was used to make a zein overcoated ingredient. A zein solution comprising 15% zein and 85% water was granulated with the polyvinyl acetate/aspartame  
30 ingredient by adding the solution slowly so as to slightly dampen the polyvinyl acetate/aspartame ingredient with the zein solution, then dried and ground. The zein overcoated ingredient was added into fruit

- 14 -

flavored chewing gum at 0.5% by weight of the gum. The gum formula as set forth below in Table 1 as Example 3.

The following table shows the gum formulas for each example. The ingredients are given in weight percentage.

5

TABLE 1

	<u>Ex. 1</u>	<u>Ex. 2</u>	<u>Ex. 3</u>
Sugar	57.43%	57.03%	57.03%
Gum Base	19.30	19.30	19.30
Dextrose Monohydrate	7.28	7.28	7.28
10 Corn Syrup	13.91	13.91	13.91
Glycerin	1.15	1.15	1.15
Fruit Flavor	0.70	0.70	0.70
Lecithin	0.13	0.13	0.13
Free Aspartame	0.10	----	----
15 PVAc Encapsulate Aspartame	----	0.50	----
Zein Overcoated Aspartame	----	----	0.50

Accelerated aging tests were conducted with the gum samples. The gum samples were placed in 85°F for 60 days. The gum samples were analyzed by HPLC to determine the stability of the aspartame over the 60 days.

20

The Figure illustrates graphically the percent loss of aspartame over time. As illustrated aspartame had the greatest stability when it was encapsulated with polyvinyl acetate and overcoated with zein (Example 3).

25

It should be understood that various changes and modifications to the presently preferred embodiments described herein will be apparent to those skilled in the art. Such changes and modifications can be made without departing from the spirit and scope of the present invention and without diminishing its attendant advantages. It is therefore intended that such changes and modifications be covered by the appended claims.

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- 15 -

WE CLAIM:

1. A chewing gum including:  
a water insoluble portion;  
one or more flavors;  
5 a water soluble portion; and  
a sensitive active ingredient coated with a first coating of polyvinyl acetate that substantially surrounds the sensitive active ingredient and a second coating of zein that substantially coats the first coating.
- 10 2. The chewing gum of Claim 1 wherein the active ingredient is chosen from the group consisting of aspartame, alitame, sucralose, and glycyrrhizin.
3. The chewing gum of Claim 1 wherein the flavor  
15 is chosen from the group consisting of amyl acetate, lemon oil, orange oil, ethyl butyrate, ethyl propionate, ethyl acetate, ethyl alcohol, ethyl caproate, and aldehyde flavors.
4. The chewing gum of Claim 1 wherein the second  
20 coating is applied at a weight ratio of about 1:10 to about 1:1 as compared to a total cumulative weight of the first coating and sensitive active ingredient.
5. The chewing gum of Claim 1 wherein the second coating is applied by wet granulation.
6. An encapsulated sensitive active ingredient  
25 comprising:  
a sensitive active ingredient;  
a first coating of polyvinyl acetate that coats at least a substantial portion of an outer surface of the sensitive active ingredient; and  
30 a second coating of zein that coats at least a substantial portion of an outer surface of the first coating.

- 16 -

7. The encapsulated sensitive active ingredient of Claim 6 wherein the active ingredient is chosen from the group consisting of aspartame, alitame, sucralose, and glycyrrhizin.

5           8. The encapsulated sensitive active ingredient of Claim 6 wherein the second coating is applied at a weight ratio of about 1:10 to about 1:1 as compared to a total cumulative weight of the first coating and the sensitive active ingredient.

10           9. The encapsulated sensitive active ingredient of Claim 6 wherein the second coating is applied by wet granulation.

15           10. The encapsulated sensitive active ingredient of Claim 6 wherein the encapsulated sensitive ingredient is used in a confectionery product.

11. The encapsulated sensitive active ingredient of Claim 6 wherein the encapsulated sensitive ingredient is used in a chewing gum.

20           12. A method for preparing chewing gum comprising the steps of:

coating a sensitive active ingredient with polyvinyl acetate to create an encapsulated product; and

25           coating the encapsulated product with a solution chosen from the group consisting of zein, gelatin and water soluble proteins.

13. The method of Claim 12 wherein the water soluble proteins are chosen from the group consisting of casein, soy, and gelatin.

30           14. The method of Claim 12 wherein the sensitive active ingredient is coated with polyvinyl acetate through wet granulation, wax granulation, spray drying, spray chilling, fluid bed coating, coacervation, extrusion, or fiber spinning.

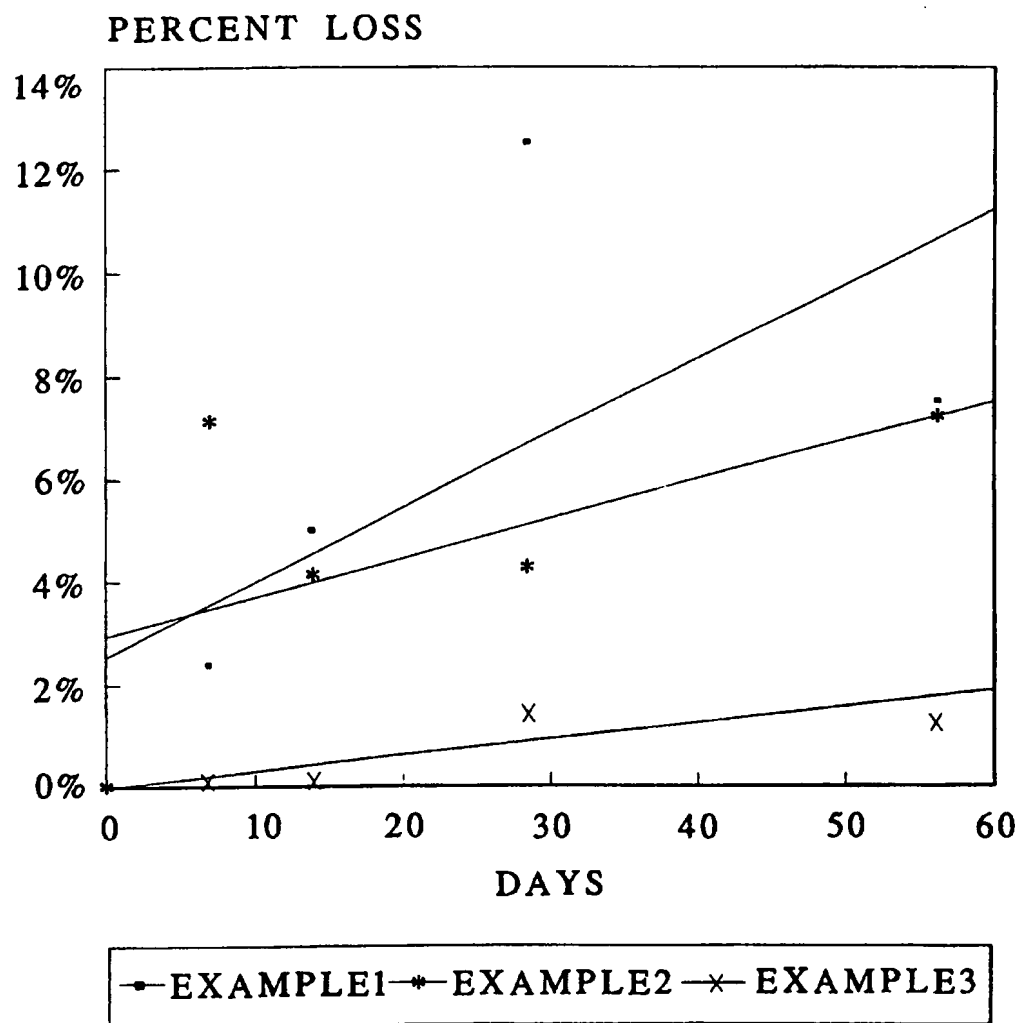
- 17 -

15. The method of Claim 12 wherein the solution is coated on the encapsulated product through wet granulation.

5 16. The method of Claim 12 wherein the solution includes zein.

17. The method of Claim 12 wherein the sensitive active ingredient is aspartame.

1/1

*FIG.1*

# INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US96/11991

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :A23G 3/30

US CL :426/5

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 426/3, 4, 5, 6, 96, 302, 310, 548

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Please See Extra Sheet.

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5,169,657 A (YATKA et al) 08 December 1992.	1-17
A	US 4,997,659 A (YATKA et al) 05 March 1991.	1-17
A	US 5,043,169 A (CHERUKURI et al) 27 August 1991.	1-17
A	US 5,139,798 A (YATKA et al) 18 August 1992.	1-17

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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Date of the actual completion of the international search 11 NOVEMBER 1996	Date of mailing of the international search report 19 DEC 1996
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# INTERNATIONAL SEARCH REPORT

International application No.

PCT/US96/11991

## B. FIELDS SEARCHED

Electronic data bases consulted (Name of data base and where practicable terms used):

APS

search terms: encapsulated aspartame, encapsulated alitame, encapsulated sucralose, encapsulated glycyrrhizin, second coating, zein